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Parameter estimations for measurements of thermal transport properties with the hot disk thermal constants analyzer

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The objective of this work is to improve measurements of transport properties using the hot disk thermal constants analyzer. The principle of this method is based on the transient heating of a plane double spiral sandwiched between two pieces of the investigated material. From the temperature increase of the heat source, it is possible to derive both the thermal conductivity and the thermal diffusivity from one single transient recording, provided the total time of the measurement is chosen within a correct time window defined by the theory and the experimental situation. Based on a theory of sensitivity coefficients, it is demonstrated how the experimental time window should be selected under different experimental situations. In addition to the theoretical work, measurements on two different materials: poly(methylmethacrylate) and Stainless Steel A 310, with thermal conductivity of 0.2 and 14 W/mK, respectively, have been performed and analyzed based on the developed theory. © *2000 American Institute of Physics.* [S0034-6748(00)03006-9]

I. INTRODUCTION

The hot disk thermal constants analyzer—sometimes referred to as the transient plane source[1] or the Gustafsson probe[2] technique—is an experimental method which has been used to produce thermal conductivity and thermal diffusivity data of a large variety of solids and liquids. One of the main advantages of this technique is the possibility to determine both the thermal conductivity and the thermal diffusivity from one single transient measurement.

The experimental arrangement of this method is based on the use of a heat source in the shape of a plane double spiral, normally referred to as a hot disk sensor, which is placed between two sample pieces. By passing an electrical current through the sensor and at the same time record the resistance increase, a measurement of the average temperature increase of the double spiral versus time is obtained, which can be used to determine the two unknown transport coefficients. Thus, the hot disk sensor acts both as a heat source and as a temperature sensor. The temperature increase in transient experiments of this kind depends on the material being studied. However, typical values of the total temperature increase are between 0.2 and 5 K.

II. THEORY

Following the solution of the thermal conductivity differential equation,[3] the mean temperature increase of the double spiral may be expressed as

$$\overline{\Delta T(\tau)} = \frac{P_0}{\sqrt{\pi^3} r \lambda} D(\tau). \tag{1}$$

Here, P_0 is the total output of power during the transient, λ is the thermal conductivity of the material under study, and $D(\tau)$ is a dimensionless time function given by

$$D(\tau) = \frac{1}{[m(m+1)]^2} \times \int_0^\tau \sigma^{-2} d\sigma \left[\sum_{l=1}^{m} l \sum_{k=1}^{m} k \times \exp\left\{ -\frac{l^2+k^2}{4m^2\sigma^2} \right\} I_0\left(\frac{lk}{2m^2\sigma^2} \right) \right], \tag{2}$$

where m is the number of spirals in the sensor and I_0 is a modified Bessel function, cf. Ref. 1.
The dimensionless time

$$\tau = \sqrt{t/\theta} \tag{3}$$

incorporates a time scale given by

$$\theta = \frac{r^2}{a}, \tag{4}$$

where t is the real time of the measurement, r is the radius of the outer concentric circle of the double spiral, and a is the thermal diffusivity of the sample. The time scale θ is referred to as the characteristic time of the transient recording.

The temperature increase is obtained from the following relation:

$$R(t) = R_0[1 + \alpha \overline{\Delta T(\tau)}], \tag{5}$$

where R_0 is the initial electrical resistance and α is the temperature coefficient of resistivity of the probe.

Starting from the above theory, the thermal transport properties can be found using an appropriate curve-fitting technique for the experimentally measured temperature versus time points. A detailed description of this experimental technique can be found elsewhere.[1] The ideal model presupposes that the double spiral sensor—assumed to consist a set of equally spaced, concentric, and circular line heat sources—is placed in a sample of infinite dimensions. In practice all real samples do have finite dimensions. However, by restricting the time of the transient, which relates to the thermal penetration depth of the transient heating, a measurement can still be analyzed as if it was performed in an infinite medium. This means that the ideal theoretical model is still valid within a properly selected time window for the evaluation.

This work, which is aimed at establishing an optimal time window for determining the desired parameters, introduces a theory of sensitivity coefficients applied to the ideal model, Eq. (1), as well as a difference analysis (sometimes named sequence analysis) of the recorded temperature versus time data. The sensitivity coefficients[4] are the theoretical foundation for determining a suitable time window to be used in the curve-fitting procedure. According to the theory behind the sensitivity coefficients, it is important to use a time window where no or low correlation exists between the two parameters—in this case the thermal conductivity and the thermal diffusivity. It is possible to determine the two parameters unambiguously if this condition is fulfilled. However, if it is not possible to select such a time window, only one parameter or a combination of parameters will be possible to estimate from the experimental data, cf. the theory of the hot plate method previously described by Kubicar and Bohac.[2]

One way to analytically find the proper time window in a transient experiment is to plot the sensitivity coefficients versus time and perform an analysis for possible linear dependence, cf. Chaps. 1 and 5 in Ref. 4. It is obvious that an analysis of this kind may indicate that the experimental conditions—especially the time scale of the transient recording—need to be revised in order to improve the experimental accuracy.

The mathematical formula for the sensitivity coefficients is given by[4]

$$\beta_p = p \frac{\partial T(x,t)}{\partial p}, \tag{6}$$

where p, in our case, is any of the thermal transport parameters and $T(x,t)$ is the temperature function given by Eq. (1). An analysis of the sensitivity coefficients reflects in what way a change in the estimated parameters influences the magnitude of the temperature response. According to the theory it is not possible to determine two transport parameters if—within a certain time range—the corresponding two sensitivity coefficients are linearly correlated. This situation can be described in the following way, with C_1 and C_2 as arbitrary constants.

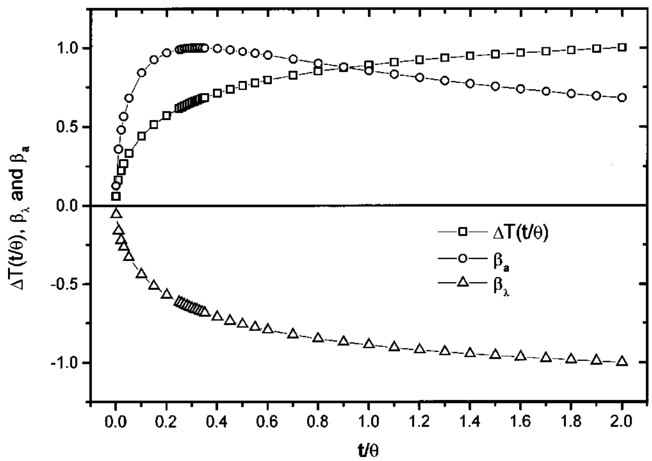

FIG. 1. Calculated temperature response and sensitivity coefficients of the two parameters—thermal conductivity β_λ and thermal diffusivity β_a. $\theta = r^2/a$ is the characteristic time of the transient experiment and t/θ is a nondimensional time scale.

$$f(t/\theta) = C_1 \beta_\alpha + C_2 \beta_\lambda. \tag{7}$$

Since we in the following will be looking at the variation of sensitivity coefficients as a function of t/θ, a more direct way of expressing a linear correlation would be as follows:

$$f(t/\theta) = C_3 \frac{t}{\theta} + C_4. \tag{8}$$

This criterion defines the time ranges where the two thermal transport parameters cannot be simultaneously and independently evaluated. For obvious reasons we are particularly interested in the time window(s) when relation (8) is not fulfilled, as we then expect to be able to evaluate the thermal conductivity and thermal diffusivity with good accuracy.

III. CALCULATION OF SENSITIVITY COEFFICIENTS

The differentiation of the temperature function, Eq. (1), for each of the two parameters, thermal conductivity and thermal diffusivity, results in two functions: the sensitivity coefficients of thermal conductivity β_λ and thermal diffusivity β_a. These coefficients are shown in Fig. 1, where the time scale is given in nondimensional form, t/θ, and the maximum values of the displayed sensitivity coefficients were normalized to 1. A straightforward analysis of the sensitivity coefficient for the thermal diffusivity shows that a maximum exists at $t/\theta = 0.33$, cf. Fig. 1. This implies that, when performing an analysis of experimental data the sensitivity of the thermal diffusivity is increasing up to this time point and decreasing from there on. This means specifically that it would be increasingly difficult to evaluate the thermal diffusivity with good precision from measurements extending over much longer times.

The absolute value of the sensitivity coefficient of the thermal conductivity is on the other hand continuously increasing, cf. Fig. 1. This means that we would be expecting better estimations of the thermal conductivity λ for longer experimental times, as long as these estimations are not affected by the process of estimating a, or vice versa. As has been pointed out above it is not possible to evaluate two

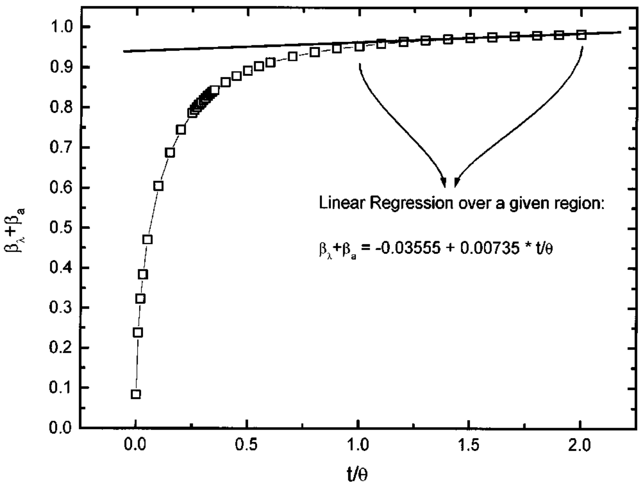

FIG. 2. The sum of the sensitivity coefficients as a function of time indicating the time window within which it should be possible to evaluate the two thermal parameters unambiguously. The data over a t/θ value greater than 1, satisfy Eq. (7) for the coefficients $C_3 = 0.007\,35$ and $C_4 = -0.035\,55$.

parameters independently from a transient recording if the sensitivity coefficients are linearly dependent within the time window of the evaluation. A linear dependence of the sensitivity coefficients has been found for $t/\theta > 1.0$ and is depicted in Fig. 2.

The theory of parameter estimation is here applied for the temperature function $\Delta T(t/\theta)$ which, according to the ideal model for the hot disk technique, is defined as an integral function from time zero to time τ, cf. Eq. (2). This means that from the sensitivity analysis above, the optimal time window starting from time zero to time $t_{\max}$, i.e., $[0,t_{\max}]$, is derived, where $t_{\max}$ is the total time of the experiment. From Figs. 1 and 2 it is clear that in the region $[0,t_{\max}]$, where $0.3 < t_{\max}/\theta < 1.1$, the sensitivity coefficients are linearly independent. An experimental assessment of this theory is provided below.

IV. DIFFERENCE ANALYSIS

When applying the theory for actual measurements it is necessary also to take nonideal experimental conditions into consideration. The first condition to be fulfilled is that the sample can be considered infinite during the transient. This condition gives an upper limit to the total time of the transient. That is, the time-dependent thermal penetration depth or the probing depth [defined as $2(at_{\max})^{1/2}$] must not be allowed to exceed the shortest distance from any point on the sensor to any point on the outer boundaries of the sample. The second nonideal condition is created by the presence of the insulating layers of the sensor, which inevitably limits the use of very short times of the transient. The minimum time $t_{\min}$ must always be chosen greater than $(\delta_{\text{ins}})^2/a_{\text{ins}}$, where δ_{ins} is the thickness and a_{ins} is the thermal diffusivity of the sensor insulation material. This minimum time corresponds experimentally to the settling time of the temperature gradients within the sensor.

The transient recordings from two materials: perspex and stainless steel, were examined with standard and difference analysis. During the total time of a transient, 200 experimental points are recorded. The total time selected for perspex was 640 s and for stainless steel 5 s. This means that the time between the recordings of the temperature is 3.2 s for Perspex and 0.025 s for stainless steel. Kapton-insulated hot disk sensors with radii of 6.675 and 3.200 mm were used in the measurements of perspex and stainless steel, respectively.

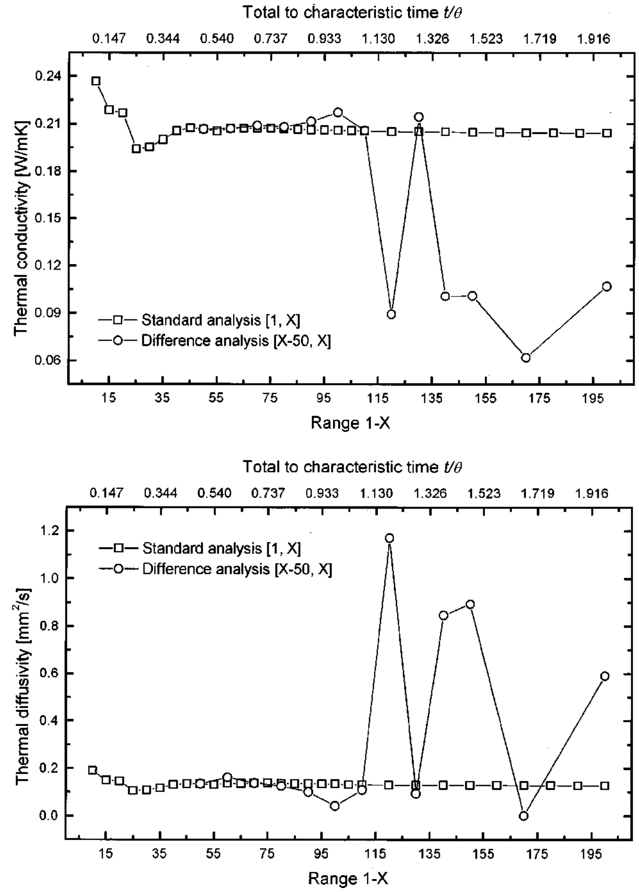

FIG. 3. The plot of thermal conductivity and thermal diffusivity as a function of time used in the fitting procedure for perspex. The standard and differential analyses show good agreement with predictions by the theoretical sensitivity coefficients.

In the standard analysis a consecutive number of points is selected for the fitting procedure, here defined as the discrete interval $[X_0, X]$. Here $(X_0 - 1)$ is the number of points deleted at the beginning of the transient and X is 200 or less. The probing depth must not exceed the shortest distance from any point on the sensor to any point on the outside surface of the sample, so the X value needs sometimes to be chosen less than 200.

The difference analysis is performed on time windows defined by the discrete intervals $[X - a, X]$, where $a = 50$ points and X ranges from point a to point 200 in steps of 5 or 10 points. This analysis gives a picture of the local sensitivity of the fit in the total time window of the measurement. Here, the experiment time was chosen to exceed the theoretical optimal time $t_{\max}$, to demonstrate the difficulties encountered to determine both transport coefficients when there exists a linear dependency of sensitivity coefficients at times $t_{\max}/\theta$ greater than 1.1. The results of the standard and 50

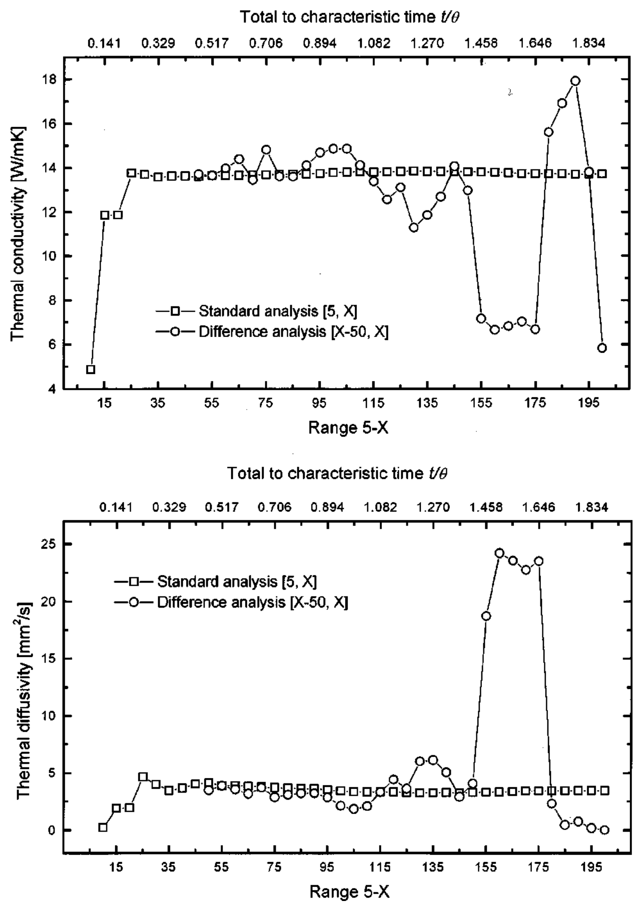

FIG. 4. The plot of thermal conductivity and thermal diffusivity as a function of time used in the fitting procedure for stainless steel. The standard and differential analyses show good agreement with predictions by the theoretical sensitivity coefficients.

point difference analysis are presented in Figs. 3 and 4, covering both thermal conductivity and thermal diffusivity results. According to these figures, the difference analysis clearly shows unstable conductivity and diffusivity results for time intervals in the vicinity of $1.1\times\theta$ and above. Thus, it can be concluded that results from the experimental difference analysis is in agreement with the results from the above theoretical sensitivity analysis. Consequently an optimal time interval for a standard analysis with the hot disk thermal constants analyzer would be $[t_{\min},t_{\max}]$, where $0.3\leqslant t_{\max}/\theta\leqslant 1.1$ and $t_{\min}\geqslant(\delta_{\rm ins})^2/a_{\rm ins}$.

V. DISCUSSION

The basic criterion for selecting experimental times in thermal conductivity experiments using the hot disk technique has been investigated. The optimal time window for determining both the thermal conductivity and the thermal diffusivity from a single transient recording has been identified as the interval $[t_{\min},t_{\max}]$ where $t_{\min}\geqslant(\delta_{\rm ins})^2/a_{\rm ins}$ and $0.3\leqslant t_{\max}/\theta\leqslant 1.1$. In this work both a theoretical analysis of the sensitivity coefficients, derived from the ideal temperature response model, as well as a difference analysis of actually performed experiments on two materials with very different thermal transport properties, verify the theoretical results and have been the basis of the final recommendations.

It is interesting to note that the experimental results from both persplex and stainless steel, displayed in Figs. 3 and 4, indicate the same range of $t_{\max}/\theta$ values for both the thermal conductivity and the thermal diffusivity. It should be pointed out that the thermal diffusivity used when calculating the characteristic time and designing the figures with the experimental results are the mean values obtained experimentally within the recommended time window.

All the experimental results, presented in this article, have been obtained with a hot disk system having a temperature resolution within the mK range. For systems with higher resolution the experimental results will naturally be more stable, however, there are so far no indications that any other time window could be recommended. The presented experimental as well as theoretical results indicate clearly that both thermal conductivity and thermal diffusivity can be determined simultaneously from one single recording, provided an appropriate time window is selected when analyzing experimental data.

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